Data Validation Checklist
Semivolatile Organic Analyses

Project:	35TH Avenue Superfund Site	Project No:	15268508.20000
Laboratory:	TestAmerica – Savannah, FL	Job ID.:	680-88767-4
Method:	SW-846 8270D (TCL SVOC)	Associated Samples:	680-88767-15 (CV0509G-CS)
Matrix:	Soil	Date(s) Collected:	03/26/2013
Reviewer:	Jane Lindsey	Date:	04/23/2013
Concurrence[1]:	Carol Lovett/Martha Meyers-Lee	Date:	04/24/2013

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
1. Were sample storage and preservation requirements met? If temperature >6°C, then J/UJ-flag results.	✓				
2. Were all COC records signed and integrity seals intact, indicating that COC was maintained for all samples?	✓				
3. Were there any problems noted in laboratory data package concerning condition of samples upon receipt?		✓			
4. Do any soil samples contain more than 50% water? If yes, then results are to be reported on a wet-weight basis.		✓			
5. Were holding times met (≤7 and 14 days from collection to extraction for aqueous and solid samples, respectively; ≤40 days from extraction to analysis)? If not, then J/UJ-flag sample results. If grossly (2x) exceeded, then flag J/R.	✓				
6. Were results for all project-specified target analytes reported?	✓				
7. Were project-specified Reporting Limits achieved for undiluted sample analyses?	✓				
8. Were samples with analyte concentrations exceeding the calibration range of the instrument re-analyzed at a higher dilution? If not, then J-flag sample result.			✓		
9. Was a method blank extracted with each batch (i.e., one per 20 samples, per batch, per matrix and per level)?	✓				
10. Were target analytes detected in the method blank?		✓			
11. Were target analytes detected in equipment/rinsate blanks?		✓		PAH were not detected during the analysis of rinsate blank 032613-RB-Shovel (680-88766-23).	
12. Are equipment/rinsate blanks associated with every sample? If		✓		According to the QAPP, a rinsate blank is to be collected after each decontamination event, which occurs once per	

[1] Independent technical reviewer

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
no, note in DV report.				week per the client. Rinsate blank 032613-RB-Shovel (680-88766-23) was collected during the week of 03/25/2013. The rinsate blank was analyzed for PAHs and metals only under Test America Job IDs 680-88766-2 and 680-88766-3, respectively. As a result, it was only possible to evaluate blank contamination for PAHs only, instead of the entire TCL SVOC list.	
13. Were analytes detected in samples below the blank contamination action level? If yes, U-flag positive sample results <5x associated blank concentration (10x for common blank contaminants – phthalates)			✓	Blank contamination does not exist.	
14. Is a field duplicate associated with this Job?		✓			
15. Was precision deemed acceptable as defined by the project plans?			✓		
16. Were DFTPP ion abundance criteria (i.e., Table 3 of SW-846 8270C) met? If no, professional judgment may be applied to determine to what extent the data may be utilized.	✓			Alternate tuning criteria were used by the laboratory (i.e., USEPA Contract laboratory Program Analytical Scope of Work). All ion abundance criteria were met per *EPA CLP National Functional Guidelines for Organic Data Review* (US EPA, October 1999).	
17. Were samples analyzed within 12 hours of the DFTPP tune? If no, professional judgment may be applied to determine to what extent the data may be utilized.	✓				
18. Were initial and continuing calibration standards analyzed at the proper frequency for each instrument? • Ensure that a minimum of five standards are used for the initial calibration. If no, use professional judgment to determine the effect on the data and note in the reviewer narrative. • An initial calibration is to be associated with each sample analysis. • A continuing calibration standard is to be analyzed for every 12 hours of sample analysis per instrument.	✓			• Initial Calibration: 04/03/2013, instrument MSG • ICV: 04/03/2013 @ 15:29 • CCV: 04/05/2013 @ 12:45	
19. Were calibration results within laboratory/project specifications? • ICAL (Criteria: ≤15 mean %RSD with individual CCC %RSD ≤30 (≤50% for poor performers), OR r≥0.995, OR r^2≥0.99, and RRF ≥0.050 (≥0.010 for poor performers)):		✓		• ICAL of 04/03/2013, instrument MSG (Lab: ≤20%RSD, Project: ≤15%RSD (≤50%RSD for poor performers)): Benzaldehyde[2] @ 48.1%RSD. Qualification of the benzaldehyde result in sample	

[2] Poor performer

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
○ If %RSD>15 (>50% for poor performers), or r <0.995, or r^2 <0.995, then J-flag positive results and UJ-flag non-detects ○ If mean RRF <0.050 (<0.010 for poor performers), then J-flag positive results and R-flag non-detects • ICV and CCV (Criteria: ≤20%D (≤50% for poor performers) and RF ≥0.050 (≥0.010 for poor performers)): ○ If %D>20 (>50% for poor performers), then J-flag positive results and UJ-flag non-detects ○ If RF <0.050 (<0.010 for poor performers), then UJ-flag non-detected semivolatile target compounds				680-88767-15 (CV0509G-CS) is not required, as the analyte is a poor performer and the %RSD is less than 50. • ICV of 04/03/2013 @ 15:29 (Lab: ≤30.0, Project: ≤20 (≤50%D for poor performers)): Benzaldehyde[2] @ -54.2%D. Positive bias is indicated by the ICV percent difference; therefore, qualification of the associated ND sample result[3] is not warranted.	
20. Was a LCS prepared for each batch and matrix?	✓				
21. Were LCS recoveries within lab control limits? If no, J-flag positive results when %R >Upper Control Limit (UCL) and J/R-flag results when %R <Lower Control Limit (LCL).	✓				
22. Were LCS/LCSD RPD within lab specifications? If no, J-flag positive results and UJ-flag non-detects			✓	LCS only	
23. Was a MS/MSD pair extracted at the proper frequency (one per 20 samples per batch)?	✓			Prep Batch 271424: • 680-88767-15 (CV0509G-CS), MS/MSD • 680-88764-3 (Batch sample), MS/MSD. According to Case Narrative, batch sample 680-88764-3 was prepared under Prep Batch 271424. The batch sample was not listed in the "GC/MS Semi VOA Batch Worksheet" on page 119 of the data package.	
24. Is the MS/MSD parent sample a project-specific sample?	✓			See above.	
25. Were MS/MSD recoveries within laboratory/project specifications? *Only QC results for project samples that are reported under this Job ID are evaluated.* • If the native sample concentration > 4x spiking level, then an evaluation of interference is not possible. • If either MS or MSD recovery meets control limits, qualification of data is not warranted. • MS and MSD %R<10: J and R Flag positive and ND results, respectively • MS and MSD %R >10 and <LCL: J-Flag positive and UJ-flag non-detect results			✓	CV0509G-CS (680-88767-15): The laboratory did not use the correct spiking solution, and MS/MSD results were not reported by the laboratory. Therefore, an evaluation is not possible.	

[3] 680-88767-15 (CV0509G-CS)

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
• MS and MSD R% >UCL (or 140): J-Flag positive results					
26. Were laboratory criteria met for precision during the MS/MSD analysis? *Only QC results for project samples that are reported under this Job ID are evaluated.* • If the native sample concentration > 4x spiking level, then an evaluation of interference is not possible. • If %RPD > UCL, J-flag positive result and UJ-flag non-detect result			✓	CV0509G-CS (680-88767-15): The laboratory did not use the correct spiking solution, and MS/MSD results were not reported by the laboratory. Therefore, an evaluation is not possible.	
27. Were surrogate recoveries within lab/project specifications? • If %R for 1 Acid or BN surrogates <10, then J-flag positive and R-flag non-detect associated sample results (i.e., acid or BN results) • If 2 or more Acid or BN %R >UCL, then J-flag positive results (i.e., acid or BN results) • If 2 or more Acid or BN %R ≥10%, but <LCL, then J-flag positive results and UJ-flag non-detect results (i.e., acid or BN results) • If 2 or more Acid or BN , with 1 %R >UCL and 1 %R ≥10%, but <LCL, then J-flag positive results and UJ-flag non-detect results (i.e., acid or BN results)	✓				
28. Were internal standard (IS) results within lab/project specifications? • If IS area counts are less than 50% of the midpoint calibration standard, then J-flag positive and UJ-flag non-detect associated sample results • If IS area counts are greater than 100% of the midpoint calibration standard, then J-flag positive results • If extremely low area counts are reported or performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated, J-flag positive and R-flag non-detect results • If retention time of sample's internal standard is not within 30 seconds of the associated calibration standard, R-flag associated data. • The chromatographic profile for that sample must be examined to determine if any false positives or negatives exists. For shifts of large magnitude, the reviewer may consider partial or total rejection of the data for that sample	✓				

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
fraction. Positive results need not be qualified as R, if mass spectral criteria are met.					
29. Were lab comments included in report?	✓			Refer to **Attachment A** (Case Narrative)	
Comments: The data validation was conducted in accordance with the *Non-Industrial Use Property Sampling Event QAPP for the 35th Avenue Removal Site, Birmingham, Alabama, Revision 1* (OTIE, October 2012). The data review process was modeled after the *USEPA Contract Laboratory Program (CLP) National Functional Guidelines (NFG) for Organic Methods Data Review* (EPA, October 1999) and *USEPA CLP NFG for Low Concentration Organic Methods Data Review* (EPA, June 2001). Sample results have been qualified based on the results of the data review process (**Attachment B**). Criteria for acceptability of data were based upon available site information, analytical method requirements, guidance documents, and professional judgment.					

DV Flag Definitions:

J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
R The sample results are unusable. The analyte may or may not be present in the sample.
U The analyte was analyzed for, but was not detected above the associated level; blank contamination may exist.
UJ The analyte was not detected above the limit, and the limit is approximate and may be inaccurate or imprecise.

ATTACHMENT A

CASE NARRATIVE

CASE NARRATIVE

Client: Oneida Total Integrated Enterprises LLC

Project: 35th Avenue Superfund Site

Report Number: 680-88767-4

With the exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. In addition all laboratory quality control samples were within established control limits, with any exceptions noted below. Each sample was analyzed to achieve the lowest possible reporting limit within the constraints of the method. In some cases, due to interference or analytes present at high concentrations, samples were diluted. For diluted samples, the reporting limits are adjusted relative to the dilution required.

Calculations are performed before rounding to avoid round-off errors in calculated results.

All holding times were met and proper preservation noted for the methods performed on these samples, unless otherwise detailed in the individual sections below.

RECEIPT

The samples were received on 03/28/2013; the samples arrived in good condition, properly preserved and on ice. The temperature of the coolers at receipt was 1.4 C.

SEMIVOLATILE ORGANIC COMPOUNDS (SOLID)

Sample CV0509G-CS (680-88767-15) was analyzed for Semivolatile Organic Compounds (Solid) in accordance with EPA SW-846 Method 8270D. The samples were prepared on 04/01/2013 and analyzed on 04/05/2013.

Method(s) 8270D: The following analytes have been identified, in the reference method and/or via historical data, to be poor and/or erratic performers: Famphur, 1,4-Napthaquinone, Methane sulfonate, Benzaldehyde, 1-naphthylamine, 2-naphthylamine, p-Dimethylamino azobenzene, p-phenylenediamine, a,a-dimethylphenethylamine, Methapyriline, 2-picoline (2-methylpyridine), 3,3'-dimethylbenzidine, 3,3'-dichlorobenzidine, Benzidine, Benzaldehyde, Benzoic acid, Dinoseb, Hexachlorophene, Hexachlorocyclopentadiene, o,o,o-triethylphosphoro-thioate. These analytes may have a %D >60% if the average %D of all the analytes in the continuing calibration verification (CCV) is 30%.

Method(s) 8270D: The initial calibration curve and initial calibration verification (ICV) analyzed in batch 272296 was outside method criteria for the following analyte(s): benzaldehyde, a,a-dimethylphenylamine, 1,4-phenylenediamine, 1-naphthylamine, hexachlorophene, and 3-methylcholanthrene. As indicated in the reference method, sample analysis may proceed; however, any detection for the affected analyte(s) is considered estimated.

Method(s) 8270D: The continuing calibration verification (CCV) analyzed in batch 272369 exceeded the method criteria for the following analyte(s): Benzaldehyde. A CCV standard at or below the reporting limit (RL) was analyzed with the affected samples and found to be acceptable. As indicated in the reference method, sample analysis may proceed; however, any detection for the affected analyte(s) is considered estimated.

MS/MSD for sample CV0509G-CS (680-88767-15) was spike with AP9 analyte solution instead of our routine 8270D spike solution. Analytes are not being reported, therefore recoveries are not calculated. Summary form III could not be generated as the compounds of concern were not spiked. Sample 680-88764-3 was also spiked in the prep batch and is included in the data set.

No difficulties were encountered during the semivolatiles analysis.

All quality control parameters were within the acceptance limits.

METALS (ICP)

Samples CV0509F-CS (680-88767-14), CV0509O-CS (680-88767-24), CV0509T-CS (680-88767-29), CV0509T-CSD (680-88767-30), CV0509Y-CS (680-88767-35), CV0509AL-GS (680-88767-52) and CV0509Y-CS (sieve) (680-88767-55) were analyzed for Metals (ICP) in accordance with EPA SW-846 Method 6010C. The samples were prepared on 03/29/2013 and analyzed on 04/02/2013 and 04/03/2013.

Samples CV0509Y-CS (680-88767-35)[2X] and CV0509Y-CS (sieve) (680-88767-55)[2X] required dilution prior to analysis. The reporting limits have been adjusted accordingly.

Several analytes recovered outside the recovery criteria for the MS/MSD of sample CV0509F-CS (680-88767-14) in batch 680-271678. Also, Chromium exceeded the rpd limit.

The presence of the '4' qualifier in the data indicates analytes where the concentration in the unspiked sample exceeded four times the spiking amount.

No other difficulties were encountered during the metals analyses.

All other quality control parameters were within the acceptance limits.

TOTAL MERCURY

Samples CV0509F-CS (680-88767-14), CV0509O-CS (680-88767-24), CV0509T-CS (680-88767-29), CV0509T-CSD (680-88767-30), CV0509Y-CS (680-88767-35), CV0509AL-GS (680-88767-52) and CV0509Y-CS (sieve) (680-88767-55) were analyzed for total mercury in accordance with EPA SW-846 Method 7471B. The samples were prepared and analyzed on 03/29/2013.

No difficulties were encountered during the mercury analyses.

All quality control parameters were within the acceptance limits.

Report revised 4/22/2013 to include case narrative comments regarding the MS/MSD data for 680-88767-15, and to remove case narrative comments about an analytical batch that was not associated with the sample data set.

ATTACHMENT B

QUALIFIED SAMPLE RESULTS

FORM I
GC/MS SEMI VOA ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah | Job No.: 680-88767-4

SDG No.: 68088767-4

Client Sample ID: CV0509G-CS | Lab Sample ID: 680-88767-15

Matrix: Solid | Lab File ID: gd0529.d

Analysis Method: 8270D | Date Collected: 03/26/2013 09:58

Extract. Method: 3546 | Date Extracted: 04/01/2013 18:43

Sample wt/vol: 30.19(g) | Date Analyzed: 04/05/2013 21:35

Con. Extract Vol.: 1(mL) | Dilution Factor: 1

Injection Volume: 1(uL) | Level: (low/med) Low

% Moisture: 29.9 | GPC Cleanup:(Y/N) N

Analysis Batch No.: 272369 | Units: ug/Kg

CAS NO.	COMPOUND NAME	RESULT	Q	RL	MDL
98-86-2	Acetophenone	470	U	470	40
1912-24-9	Atrazine	470	U	470	33
100-52-7	Benzaldehyde	470	U	470	82
92-52-4	1,1'-Biphenyl	470	U	470	1000
111-91-1	Bis(2-chloroethoxy)methane	470	U	470	55
111-44-4	Bis(2-chloroethyl)ether	470	U	470	64
108-60-1	bis (2-chloroisopropyl) ether	470	U	470	43
117-81-7	Bis(2-ethylhexyl) phthalate	220	J	470	41
101-55-3	4-Bromophenyl phenyl ether	470	U	470	51
85-68-7	Butyl benzyl phthalate	470	U	470	37
105-60-2	Caprolactam	470	U	470	94
86-74-8	Carbazole	80	J	470	43
106-47-8	4-Chloroaniline	940	U	940	74
59-50-7	4-Chloro-3-methylphenol	470	U	470	50
91-58-7	2-Chloronaphthalene	470	U	470	50
95-57-8	2-Chlorophenol	470	U	470	57
7005-72-3	4-Chlorophenyl phenyl ether	470	U	470	62
91-94-1	3,3'-Dichlorobenzidine	940	U	940	40
120-83-2	2,4-Dichlorophenol	470	U	470	50
84-66-2	Diethyl phthalate	470	U	470	52
105-67-9	2,4-Dimethylphenol	470	U	470	62
131-11-3	Dimethyl phthalate	470	U	470	48
84-74-2	Di-n-butyl phthalate	470	U	470	43
534-52-1	4,6-Dinitro-2-methylphenol	2400	U	2400	240
51-28-5	2,4-Dinitrophenol	2400	U	2400	1200
121-14-2	2,4-Dinitrotoluene	470	U	470	69
606-20-2	2,6-Dinitrotoluene	470	U	470	60
117-84-0	Di-n-octyl phthalate	470	U	470	41
118-74-1	Hexachlorobenzene	470	U	470	55
87-68-3	Hexachlorobutadiene	470	U	470	51
77-47-4	Hexachlorocyclopentadiene	470	U	470	58
67-72-1	Hexachloroethane	470	U	470	40
78-59-1	Isophorone	470	U	470	47
95-48-7	2-Methylphenol	470	U	470	38

FORM I
GC/MS SEMI VOA ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah | Job No.: 680-88767-4

SDG No.: 68088767-4

Client Sample ID: CV0509G-CS | Lab Sample ID: 680-88767-15

Matrix: Solid | Lab File ID: gd0529.d

Analysis Method: 8270D | Date Collected: 03/26/2013 09:58

Extract. Method: 3546 | Date Extracted: 04/01/2013 18:43

Sample wt/vol: 30.19(g) | Date Analyzed: 04/05/2013 21:35

Con. Extract Vol.: 1(mL) | Dilution Factor: 1

Injection Volume: 1(uL) | Level: (low/med) Low

% Moisture: 29.9 | GPC Cleanup:(Y/N) N

Analysis Batch No.: 272369 | Units: ug/Kg

CAS NO.	COMPOUND NAME	RESULT	Q	RL	MDL
15831-10-4	3 & 4 Methylphenol	460	J	470	61
88-74-4	2-Nitroaniline	2400	U	2400	64
99-09-2	3-Nitroaniline	2400	U	2400	65
100-01-6	4-Nitroaniline	2400	U	2400	69
98-95-3	Nitrobenzene	470	U	470	37
88-75-5	2-Nitrophenol	470	U	470	58
100-02-7	4-Nitrophenol	2400	U	2400	470
621-64-7	N-Nitrosodi-n-propylamine	470	U	470	45
86-30-6	N-Nitrosodiphenylamine	470	U	470	47
87-86-5	Pentachlorophenol	2400	U	2400	470
108-95-2	Phenol	470	U	470	48
95-95-4	2,4,5-Trichlorophenol	470	U	470	50
88-06-2	2,4,6-Trichlorophenol	470	U	470	41

CAS NO.	SURROGATE	%REC	Q	LIMITS
321-60-8	2-Fluorobiphenyl	74		58-130
367-12-4	2-Fluorophenol (Surr)	44		40-130
4165-60-0	Nitrobenzene-d5 (Surr)	71		46-130
4165-62-2	Phenol-d5 (Surr)	66		49-130
1718-51-0	Terphenyl-d14 (Surr)	76		60-130
118-79-6	2,4,6-Tribromophenol (Surr)	67		58-130